IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re patent application of

Piana & Gaston Confirmation No. 9268

Serial No. 10/723,828 Group Art Unit 1762

Filed November 26, 2003 Examiner Erma Cameron

For METHODS, SYSTEMS AND COMPOSITIONS FOR FIRE RETARDING SUBSTRATES

Commissioner for Patents
PO Box 1450
Alexandria, Virginia 22313-1450

DECLARATION OF SANG-HOON LIM
UNDER 37 C.F.R. §1.132

Sang-Hoon Lim declares as follows:

1. I am an expert in the textile field and am qualified to provide expert opinion on matters pertaining to textile dye operations and application of materials such as dyes and flame retardant chemicals to textiles. I have the degrees of M.S. in textile chemistry and Ph.D. in fiber and polymer science from the College of Textiles at North Carolina State University. My undergraduate degree was a B.E. in dyeing and finishing from Kyungpook National University. I am an author on a number of papers published in notable journals in the trade. As evidence of my expertise I attach my resume hereto as Attachment 1.

2. Currently I am employed by Tintoria Piana U.S., Inc., which is an owner of the above-identified application. I have additional work experience on textile products at a commercial manufacturer, and I have worked as a post-doctoral research associate at the college of textiles at North Carolina State University, and as a research assistant at the dyeing and finishing laboratory at Kyungpook National University. This background and experience has familiarized me with an array of different types of equipment and procedures used with textile products.

3. I have read and understand the above-identified patent application. I have also read and understand U.S. Patent 5,156,890 to Rock, and the office action mailed February 7, 2006.

4. Briefly described, the invention described in U.S. Patent Application 10/723,828 pertains to the application of flame retardant compounds to fibers and other textile products in a manner which provides the advantages of (1) providing a consistent amount of flame retardant compounds to the fibers or textile products run-to-run such that each batch of fibers or textile products has the same fire retardant properties, (2) eliminating waste by permitting re-use of fire retardant composition on multiple batches of fibers or textile products (this having the added advantage of being environmentally friendly), and (3) improving the throughput (speed) of multiple batches of fibers or textile products. With reference to Figure 1 of the application, it can be seen that one embodiment of the invention is to use commercial dye machines 32 and 34 in a "closed loop system" which includes a variety of pipes, valves, mixing tanks, centrifuges, and flow controllers. As can be seen from Figure 1, and as is described in the patent application, fire retardant composition which is not absorbed into fibers in dye machine 32 is sent to dye machine 34 (and vice versa). Further, fire retardant composition extracted from the fibers or textile products by centrifuges 12 and 14, is also re-used in the "closed loop system" and can be sent to either of the dye machines 32 and 34 along pathways leading to the mixing tanks 28 and 30. In this way, the fire retardant composition is recycled and reused repetitively and essentially infinitely. As fire retardant composition is "depleted" (i.e., a certain amount of water and fire retardant compound will be absorbed into the fiber), additional chemicals and water can be added in the closed loop system to replenish the amounts lost (equaling the amount "depleted" (i.e., absorbed)) in each process step (see items 66, 84, 90, 94, 98, 102, and 108 in the drawing figure).

5. In the office action dated February 7, 2006, it has been concluded that "the admitted state of the prior art is that closed loop dyeing machines for fibers are known and are available from sources known in the art (71:20-72:12)". This conclusion is not correct. In particular, pages 71:20-72:12 make no such as admission, and the conclusion the "closed loop dyeing machines for fibers are known and are available" is not correct.

Page 71 of the application identifies a number of known dyeing machines. None of the dyeing machines on page 71 are "closed loop dyeing machines for

fibers". Dyeing fibers is completely different from applying fire retardant chemicals to fibers. All dyes have inherent affinity for the textile fibers at least during some stage of its application. Dyes are soluble in the dyeing medium (e.g., water) and they are adsorbed on fibers and then diffuse into the fibers. This phenomenon is referred to as "exhaustion". See for example, Textile Coloration and Finishing, Warren S. Pertkins, Carolina Academic Press, January 1996, ISBN: 0890898855. Due to the dye exhaustion into fibers, the dye concentration in the water at the beginning decreases significantly at the end of dyeing. The remaining dye solution is drained as waste in a normal dyeing process. This is done for a variety of reasons, but it should be clear that the dye composition changes (i.e., the concentration of dye in the medium decreases) such that subsequent batches of fibers would not be effectively dyed the same by re-using the dye and would not have the same appearance or other qualities desired in textile processing.

A few flame retardant compounds can be applied by exhaustion as discussed above (especially for polyester or polyamide fibers). However, fire retardant compounds are not "exhausted" into cellulosic fibers such as cotton and rayon because there is no inherent affinity between fire retardant compounds and cellulosic fibers. In applying fire retardant compounds to cellulosic fibers or textile products, the fiber or textile product is saturated with aqueous fire retardant solution. As discussed in Textile Finishing, edited by Derek Heywood, published by the Society of Dyers and Colourists, 2003, ISBN: 0901956813, the normal fire retardant treatment method for cellulosic fibers is by soaking and drying (this was also considered "normal" at the time the invention was made). The concentration of the fire retardant composition in water does not change at the end of the treatment, because the treatment is by saturation, not exhaustion.

Fire retardant compounds are often applied to cellulosic fiber by spraying the fire retardant solution on the fiber or by soaking fibers in the solution, and then drying them. However, prior methods do not ensure an even distribution of fire retardant composition throughout the fiber, thus these treatments generally result in uneven fire retardant performance from run-to-run, and even within the same run.

In the present patent application, a "closed loop" process is described which can employ "components" of conventional dye machines (see page 72 at line 6). The "closed loop system" is entirely new (page 72 of the application

describes application of the components of dye machines in the "invention"; thus, it should be understood that the closed loop system is new and is not used in dye machines of the prior art) and required modification and adaptation of pumps, valves, cycle programs, flow pathways, etc. The patent specification provides the recognition that, in a preferred embodiment of the invention, a stock dyeing machine can be used to ensure strong circulation of fire retardant solution throughout a batch of fibers. Other devices might also be used to ensure circulation of the fire retardant solution throughout the fibers. However, it should be understood that, prior to the invention by the applicants, there were no commercially available dyeing machines configured in a "closed loop system" as contemplated by the patent application.

In the present application, the used fire retardant solution is collected and re-used, either on the same batch of fibers or on subsequent batches of fibers. For a subsequent treatment, one simply adds the amount of fresh fire retardant composition that equals the amount used by the previous treatment. The used amount is the amount of absorbed fire retardant solution on fibers, and this is around 80% by weight of the fiber weight (depending on the fiber and other factors). This closed loop system is possible because the flame retardant treatment on cellulosic fiber is by saturation, which does not alter the concentration of the fire retardant solution before and after treatment.

6. One of ordinary skill in the art of textile processing would typically have a bachelors degree or higher in textiles, chemistry or chemical engineering, and would have knowledge of chemistry and physics so as to recognize differences between exhaustion processes used in dyeing and saturation processes used in application of flame retardant compounds. He or she may have five to ten years experience, and would have access to basic texts on textile processing. Examples of such texts may include those discussed above, and <u>Cellulosics Dyeing</u>, edited by John Shore, published by the Society of Dyers and Colourists, 1995, and <u>Textile Processing and Properties: Preparation, Dyeing, Finishing and Performance</u>, Tyrone L. Vigo, Published by Elsevier Science, 1994. He or she would recognize from reading the patent application, particularly pages 94 to 110, that exhaustion is not contemplated by the invention, but that the invention contemplates adding back (replenishing) amounts of fire retardant composition

which are depleted in each run (i.e., absorbed into the fiber). The "closed loop system" shown in Figure 1 of the application shows a variety of mechanisms for replenishing the amounts of fire retardant composition which are absorbed into the fibers (e.g., movement of fire retardant composition that is not absorbed directly between the dye machines 32 and 34 through pipes and valves (46, 48, 50, 52, 54, 56, 58, and 60); recovery of fire retardant composition from the fibers through centrifugation (12 and 14) and addition of the collected amounts to collection tank 20; and preparation of the fire retardant composition from chemicals (90, 94, and 102) and water (84, 100, and 108)). Thus, in my opinion, the patent application fully describes the application in a manner would allow one of ordinary skill in the art to make and use the invention.

7. In my opinion, U.S. Patent 5,156,890 to Rock describes retrofitting conventional laundry equipment so that fire retarding chemicals can be applied to fabrics such as denim pants. Rock envisions that one can re-apply fire retardant to denim pants with each wash cycle. As such, Rock is not contemplating having the fiber of the denim pants retain flame retardant property after being washed, laundered or dry cleaned. Instead, Rock describes a system where one simply re-applies fire retardant compounds to the fabric. In particular, Rock describes applying fire retardant compounds to <u>damp</u> clothing after it has been laundered. Thus, the damp clothing already contains water, and when the fire retardant composition is applied, there is necessarily dilution of the fire retardant composition (a reduction in concentration) by the water already present in the clothes. As such, Rock does not contemplate a system where the concentration of fire retardant compounds remains constant from run to run. In addition, Rock is focused on treating manufactured clothing, not fibers. Rock utilizes the agitation provided by the washing machine, and does not contemplate circulating fire retardant composition through a stack of stationary fibers, as would occur in a dye machine. Furthermore, Rock does not describe and would not suggest to one skilled in the art a closed loop system where fire retardant chemicals are, for example, retrieved from one dye machine and used on a different batch of fibers in a different dye machine. Rock shows a batch process where after one load of laundry is done, the next load is performed.

8. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: May 31, 2006

Sang-Hoon Lim

SANG-HOON LIM

SUMMARY

- MS, Ph.D., and Post-doctoral research experience from College of Textiles, NC State Univ, which is the best textile school in the US.
- Solid textile knowledge: Major in textile chemistry, which includes polymer science, dye chemistry, color science, textile materials (natural and man-made fiber, yarn and fabric formation), and textile wet-processes (preparation, dyeing, and finishing).
- Direct experience in the development and improvement of high-performance textile finishes, which include water/oil repellent, permanent press, antimicrobial, odor resistance, flame retardant, and aroma finishing.
- Utilization of natural resources for textile application: Use of chitosan for textile dyeing and finishing.
- Development of energy saving textile bleaching system.
- Statistical design of experimental (DOE) for process optimization.
- Competent in a broad range of scientific disciplines, which include organic chemistry, polymer chemistry, and analytical chemistry.

EDUCATION

North Carolina State University **Raleigh, NC**
Ph. D. in Fiber and Polymer Science (2003)
Synthesis of a Fiber-Reactive Chitosan Derivative and Its Application to Cotton Fabric as an Antimicrobial Finish and a Dyeing-Improving Agent. (Advisor: Prof. Samuel M. Hudson).

Master of Science in Textile Chemistry (1999)
Synthesis and Characterization of a Fiber-Reactive and Water-Soluble Chitosan Derivative with Enhanced Antimicrobial Activity. (Advisor: Prof. Samuel M. Hudson).

<u>GPA: 4.0/4.0</u>

Kyungpook National University **Daegu, Korea**
Bachelor of Engineering in Dyeing and Finishing (1996)

<u>GPA: 4.05/4.3</u>

RESEARCH & WORK EXPERIENCE

Tintoria Piana U.S., Inc. **Cartersville, GA**
Sr. R&D Engineer (Oct. 2005 ~ Present)
Research and Development for high performance and value-added textile products.
Process optimization and quality control.

Nano-Tex, Inc. **Emeryville, CA**
Associate Scientist (Oct. 2004 ~ Oct. 2005)
Research and Development for high performance and value-added textile products using innovative textile chemical finishes. (R&D in Lab and scale-up on production equipment in conjunction with manufacturing and process engineers).

North Carolina State University (College of Textiles) **Raleigh, NC**
Post-doctoral research associate (Jan. 2003 ~ Sep. 2004)
Supervisor: Dr. Peter Hauser and Dr. David Hinks
Development and commercialization of low-energy bleaching systems using cationic bleach activators. Utilization of statistical experimental design for optimization of the process.

Research assistant (May. 1998 ~ Dec. 2002)
Advisor: Prof. Samuel Hudson
Application of chitosan derivatives in textile dyeing and antimicrobial finishing.

Kyungpook National University **Daegu, Korea**
Research assistant at Dyeing and Finishing Laboratory **(Oct. 1996 ~ May 1997)**
Advisor: Prof. Yong-Jin Lim (former President of The Korean Society of Dyers and Finishers)
Jet-black dyeing of polyester. Microcapsulation of fragrances for textile application.

PUBLICATIONS

- Jung Jin Lee, Sang-Hoon Lim, Peter Hauser, and David Hinks, Synthesis of novel cationic bleach activators and their hydrolytic stabilities in aqueous solution, *article in preparation*.
- Sang-Hoon Lim, Jung Jin Lee, David Hinks and Peter Hauser, Bleaching of cotton with activated peroxide systems, *Coloration Technology*, **121**(2), 89-95 (2005).
- Jung Jin Lee, Sang-Hoon Lim, Peter Hauser and David Hinks, Stability of a novel cationic bleach activator in aqueous solution, *Coloration Technology*, **121**(1), 37-40 (2005).
- Sang-Hoon Lim, Nevin Çiğdem Gürsoy, Peter Hauser, and David Hinks, Performance of a cationic bleach activator in a hot bleaching system, *Coloration Technology*, **120**(3), 114-118 (2004).
- Nevin Çiğdem Gürsoy, Sang-Hoon Lim, David Hinks, and Peter Hauser, Evaluating Hydrogen Peroxide Bleaching with Cationic Bleach Activators in a Cold Pad-Batch Process, *Textile Research Journal*, **74**(11), 970-976 (2004).
- Sang-Hoon Lim and Samuel M. Hudson, Application of a fibre-reactive chitosan derivative to cotton fabric as a zero salt dyeing auxiliary, *Coloration Technology*, **120**(3), 108-113 (2004).
- Sang-Hoon Lim and Samuel M. Hudson, Application of a fiber-reactive chitosan derivative to cotton fabric as an antimicrobial textile finish, *Carbohydrate Polymers*, **56**(2), 227-234 (2004).
- Sang-Hoon Lim and Samuel M. Hudson, Synthesis and antimicrobial activity of a water-soluble chitosan derivative with a fiber-reactive group, *Carbohydrate Research*, **339**, 313-319 (2004).
- Sang-Hoon Lim and Samuel M. Hudson, Review of chitosan and its derivatives as antimicrobial agents and their uses as textile chemicals, *Journal of Macromolecular Science – Polymer Reviews*, **C43**(2) (2003) 223-269.
- S. Lim, K. Hattori, and S. M. Hudson, Preparation of a fiber-reactive chitosan derivative with enhanced antimicrobial activity, in Advances in Chitin Science, Vol. IV (M.G. Peter, A.

Domard and R.A.A. Muzzarelli, Eds.); Universitat Potsdam, Potsdam, Germany, 2000, pp 454-459.

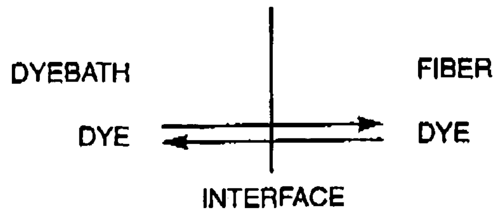
PATENTS

- Peter Hauser, David Hinks, Jung Jin Lee, and Sang-Hoon Lim, A Cationic Bleach Activator with Enhanced Hydrolytic Stability, *US Patent in Process (NCSU's intellectual Property Committee accepted the invention of management)* (2005).

ORGANIZATIONS

American Association of Textile Chemists and Colorists (Senior member)

Figure 4-1. Dyeing as a mass transfer process



dyeing process, consider the model of a dyeing system shown in Figure 4-1.

This model describes dyeing as a mass transfer process. The model is applicable to virtually all dyeing systems; continuous or batch, cotton or synthetic fibers, fiber or fabric or garments. The task that must be accomplished in dyeing is to transfer dye from the dyebath to the fiber. The term "exhaustion" is used to express the degree of dye transfer from dyebath to fiber. Exhaustion is usually expressed as a percentage of the amount of dye originally placed in the dyebath. For example, if ¾ of the dye originally added to the dyebath transfers to the fiber, the exhaustion is 75%. The depth of color achieved depends mainly on how much dye is added to the fiber. The location of dye on the fiber has some influence on the apparent depth of color. Dye close to the surface of the fiber or near the surface of the yarn or fabric contributes more to the apparent depth than does deeply penetrated dye. Thus, the apparent darkness of the dyeing is not necessarily the same for two fabrics containing exactly the same amount of dye.

For economic and environmental reasons, a high degree of exhaustion is desirable. Dyes are expensive, and dye which is left in the bath is wasted. Furthermore, dye left in the dyebath is a pollutant which must be controlled and disposed of along with the wastewater from the plant. Often, auxiliary chemicals are added to the dyebath to improve exhaustion.

Dye exhausts gradually from the dyebath to the fiber as time passes, but there is usually a diminishing return in exhaustion as time of dyeing increases. After some finite dyeing time, the additional time and chemicals required to produce higher exhaustion may be more expensive than savings achieved in dye and waste treatment costs.

Most dyeing processes are reversible. That is, as dye molecules transfer from the dyebath into the fiber, other dye molecules desorb